Declaration

I, Dr. Eduard Michel, state that I am a resident of D-60529 Frankfurt, Federal Republic of Germany; that I am a citizen of the Federal Republic of Germany; that I am a chemist having graduated at the University of Freiburg, Federal Republic of Germany; that I am one of the inventors of U.S. Patent Application Serial No. 09/722,760; for „Use of salt-like structured silicas as charge control agents"; that I consider myself qualified , by my knowledge of chemistry, and especially of electrophotographic toners and developers and by my 9 years' experience in this field; that I have made the following observations to wit:

In order to demonstrate that a tertiary ammonium molybdate which is used as charge control agent in JP8-6295-A significantly reduces the amount of negative charge of an electrophotographic toner compared with a toner according to U.S. Patent Application Serial No. 09/722,760 wherein a structured silicate salt acts as a charge control agent, the following experiments were carried out:

A) Electrophotographic toner according to U.S. Patent Application Serial No. 09/722,760

A1) Polyester resin toner:
1 part of the dimethyldistearyl ammonium bentonite of Preparation Ex. 1 was incorporated homogeneously using a kneader over the course of 45 minutes into 99 parts of a polyester resin based on bisphenol A (®Almacryl T 500). The composition was then milled on a laboratory universal mill and subsequently classified on a centrifugal screen-classifier. The desired particle fraction (4 to 25 µm) was activated using a carrier which consists of styrene-methacrylate copolymer-coated magnetite particles with a size of 50 to 200 µm .

A2) Styrene acrylate resin toner:
1 part of the dimethyldistearyl ammonium bentonite of Preparation Ex. 1 was incorporated homogeneously using a kneader over the course of 45 minutes into 99 parts of a 60:40 styrene-methacrylate copolymer toner binder (®Dialec S 309). The composition was then milled on a laboratory universal mill and subsequently classified on a centrifugal screen-classifier. The desired particle fraction (4 to 25 µm) was activated using a carrier which consists of styrene-methacrylate copolymer-coated magnetite particles with a size of 50 to 200 µm.

Measurement was carried out using a conventional q/m measurement setup. A screen with a mesh size of 50 µm was used to make sure that , when the toner was blown out, no carrier was ejected with it. Measurements were carried out at 50 % relative atmospheric humidity. The q/m values (µC/g) were measured as a function of the activation period (5 minutes, 10 minutes, 30 minutes, 2 hours):

	Charge q/m in μC/g			
	5 min	10 min	30 min	2h
A1):	- 22	- 18	- 13	- 11
A2):	-20	- 21	- 18	- 10

B) Electrophotographic toner according to JP8-6295-A:

B1): Polyester resin toner:
1 part of the dimethyldistearyl ammonium bentonite **and 1 part of alkylated quarternary ammonium molybdate** (®TP-415, Hodogaya Chemical Co.) were incorporated homogeneously using a kneader over the course of 45 minutes into 98 parts of a polyester resin based on bisphenol A (®Almacryl T 500). The composition was then milled on a laboratory universal mill and subsequently classified on a centrifugal screen-classifier. The desired particle fraction (4 to 25 μm) was activated using a carrier which consists of styrene-methacrylate copolymer-coated magnetite particles with a size of 50 to 200 μm.

B2): Styrene acrylate resin toner:
1 part of the dimethyldistearyl ammonium bentonite **and 1 part of alkylated quarternary ammonium molybdate** (®TP-415, Hodogaya Chemical Co.) were incorporated homogeneously using a kneader over the course of 45 minutes into 98 parts of a 60:40 styrene-methacrylate copolymer toner binder (®Dialec S 309). The composition was then milled on a laboratory universal mill and subsequently classified on a centrifugal screen-classifier. The desired particle fraction (4 to 25 μm) was activated using a carrier which consists of styrene-methacrylate copolymer-coated magnetite particles with a size of 50 to 200 μm.

Measurement was carried out using a conventional q/m measurement setup. A screen with a mesh size of 50 μm was used to make sure that , when the toner was blown out, no carrier was ejected with it. Measurements were carried out at 50 % relative atmospheric humidity. The q/m values (μC/g) were measured as a function of the activation period (5 minutes, 10 minutes, 30 minutes, 2 hours):

	Charge q/m in μC/g			
	5 min	10 min	30 min	2h
B1):	- 13	- 11	- 9	- 8
B2):	- 3	- 3	- 3	- 2

Results:

The Test clearly shows that an electrophotographic toner comprising an alkylated quarternary ammonium molybdate as disclosed in **JP8-6295-A, Table 1** exhibits a noticeably lower negative charge level during the entire activation period, compared to a toner according to U.S. Patent Application Serial No. 09/722,760.

Evaluation of Results:

JP8-6295-A clearly teaches to use an alkylated quarternary ammonium molybdate as a potent charge-control agent in combination with an (organic) bentonite as an effective extender for improving dispersibilty. Incorporated in above customary toner binders the resulting electrophotographic toners show a low, negative charge.

It was unpredictable that

(i) the quaternary ammonium molybdate can be omitted,
(ii) an organic bentonite can take over the function of the charge control agent, and
(iii) that the resulting charge is a significantly higher, negative charge.

Therefore, U.S. Patent Application Serial No. 09/722,760 provides for a valuable method for improving and imparting the charge of toners especially for negative-charged copy and printing machines.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Frankfurt on the Main
November 1st, 2004

Eduard Michel

(Eduard Michel)